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# The Synthesis of Hybrid Materials by the Blending of Polyhedral Oligosilsesquioxanes into Organic Polymers

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## Edwards AFRL

0 100mi  
100km

Hawthorne

San Jose

Salinas

Fresno

Bakersfield

Los Angeles

Orange

Oceanside

Phoenix

San Diego





## Hybrid Organic/Inorganic Blends

- GOAL: To study the interaction and solubility of Polyhedral Oligosilsesquioxane (POSS) molecules containing various organic side groups with the polymer matrix
- Polystyrene was chosen since it is readily available and can easily be solvent cast with the POSS molecules for TEM studies

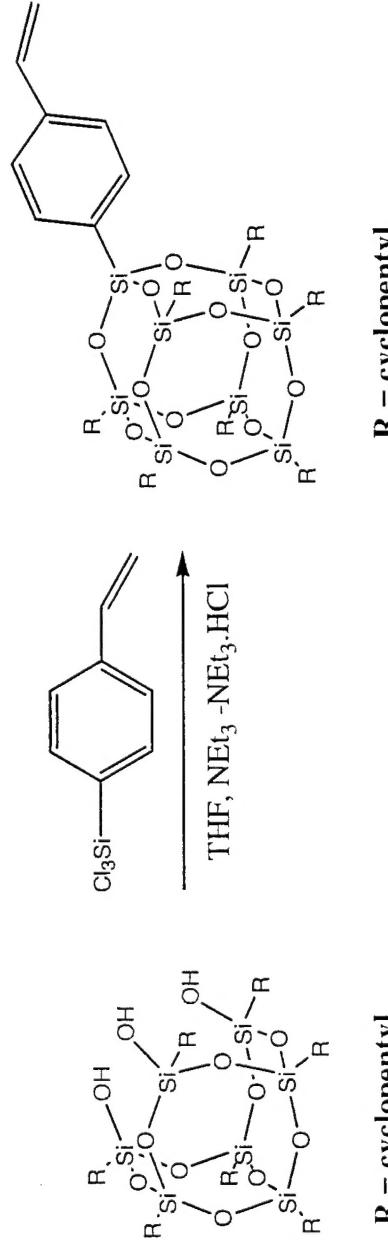
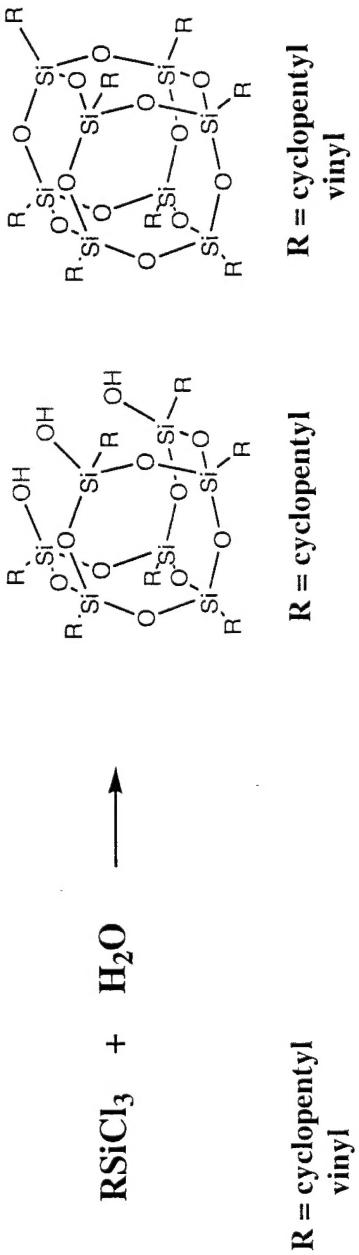


## Why Use Blendables?

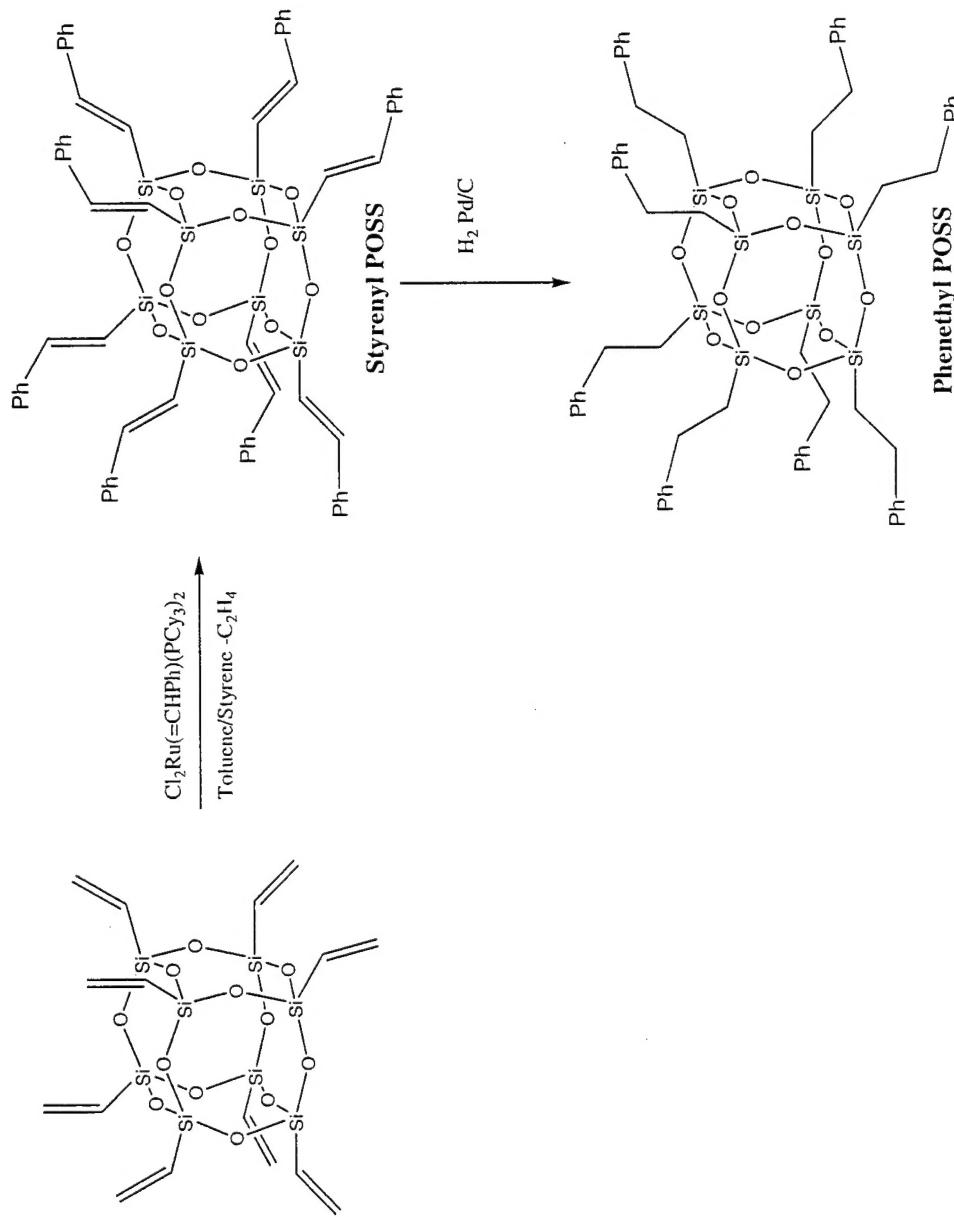
- Easier to tailor the organic side groups of the POSS molecule to give a polymer-soluble species
- Simple blending techniques can be used instead of copolymerization with reactive POSS monomers
- Potential Drop-in molecular modifier without requiring expensive replacement of processing equipment

# POSS = Polyhedral Oligomeric Silsesquioxane General Synthesis

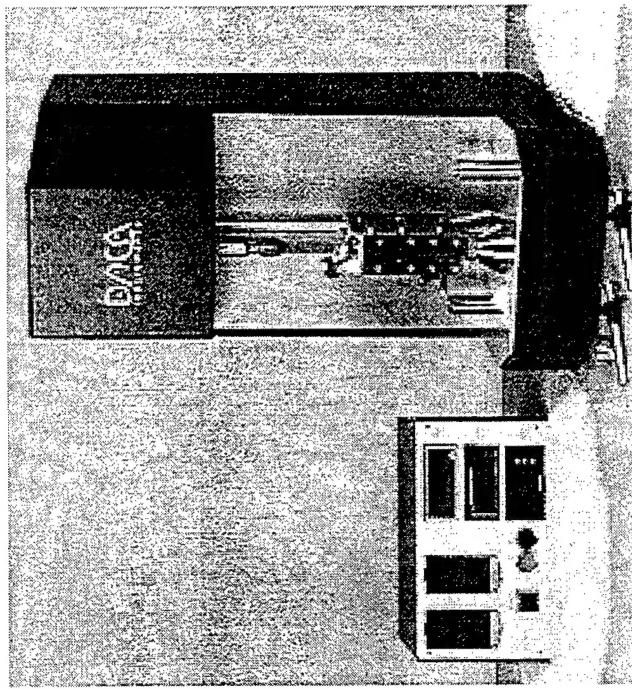
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# POSS = Polyhedral Oligomeric Silsesquioxane General Synthesis



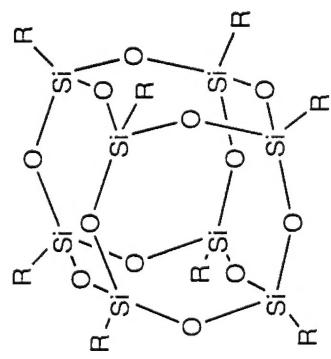
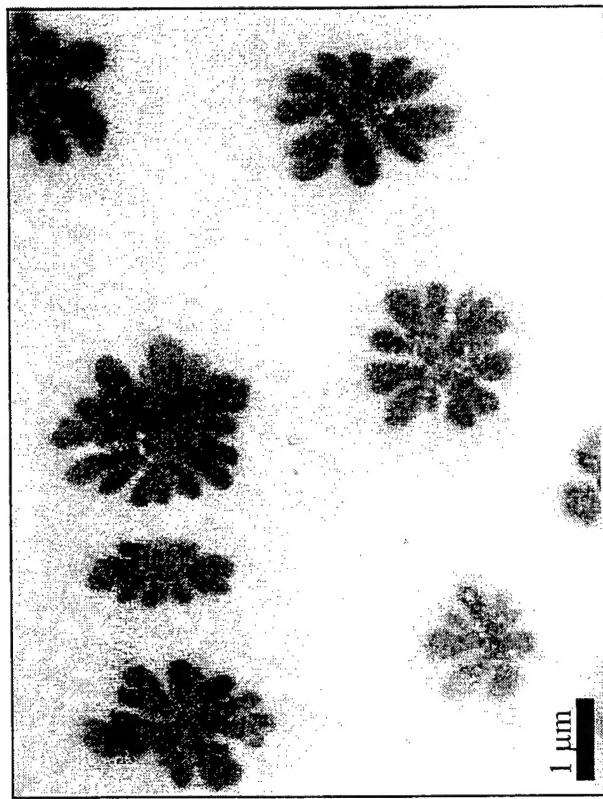
# Preparation of Styrene-POSS Blends



- TEM Method
- Dissolve the Styrene and POSS in THF
- Cast very thin film by slow solvent evaporation
- Traditional Processing
- Place Polystyrene in Extruder
- Add POSS
- Blend 2-5 Minutes

# **POSS Blends - Crystal Formation**

50 wt %  $\text{Cp}_8\text{T}_8$  in 2 million mol. wt. Polystyrene



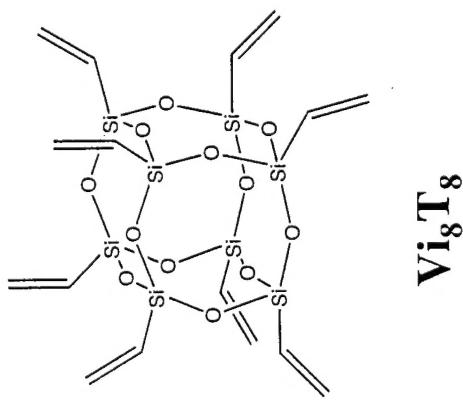
R = cyclopentyl

$\text{Cp}_8\text{T}_8$

**TEM image clearly shows formation of immiscible POSS  
crystallites (20-50k molecules)**

## POSS Blends - Crystal Formation

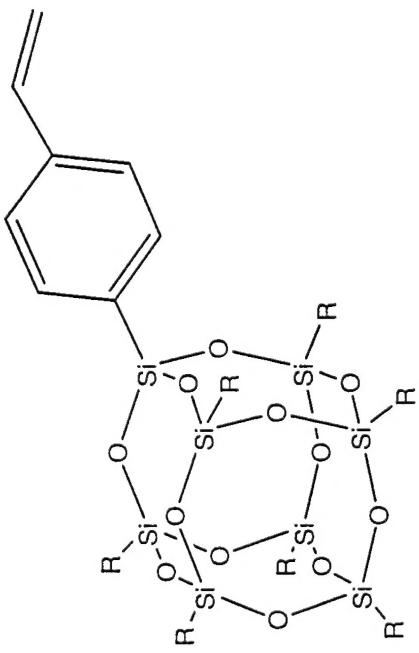
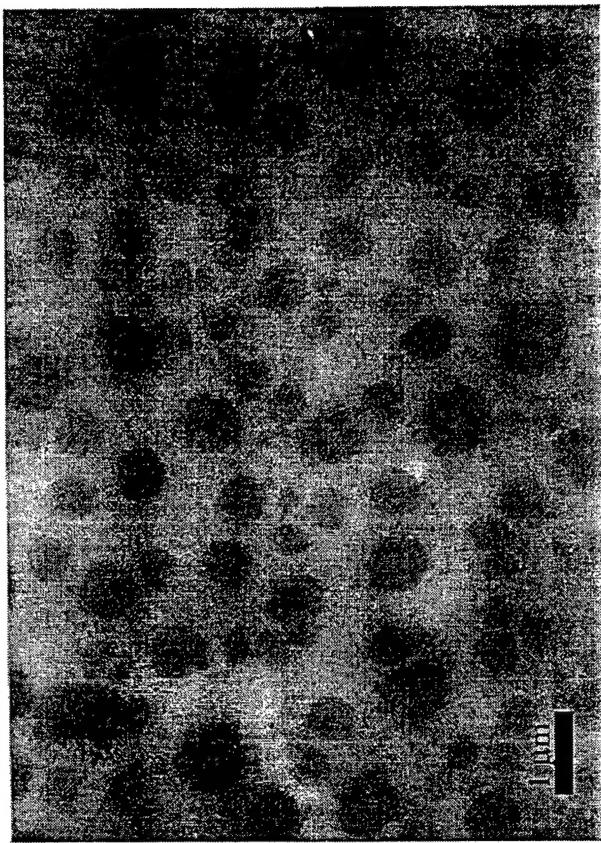
50 wt %  $\text{V}_{\text{i}}\text{Ti}_8$  in 2 million mol. wt. Polystyrene



TEM image clearly shows immiscibility in polymer system

## **POSS Blends - Increased Solubility**

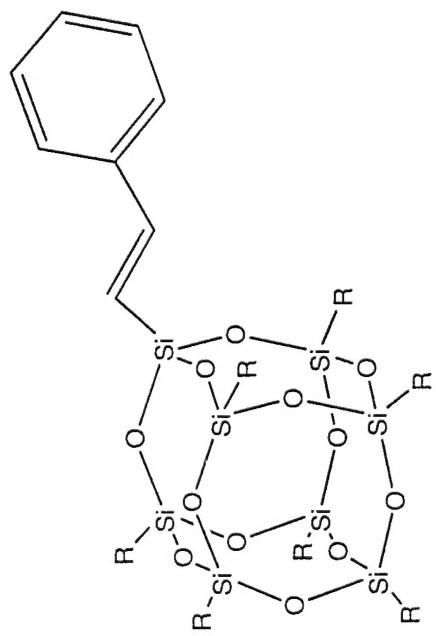
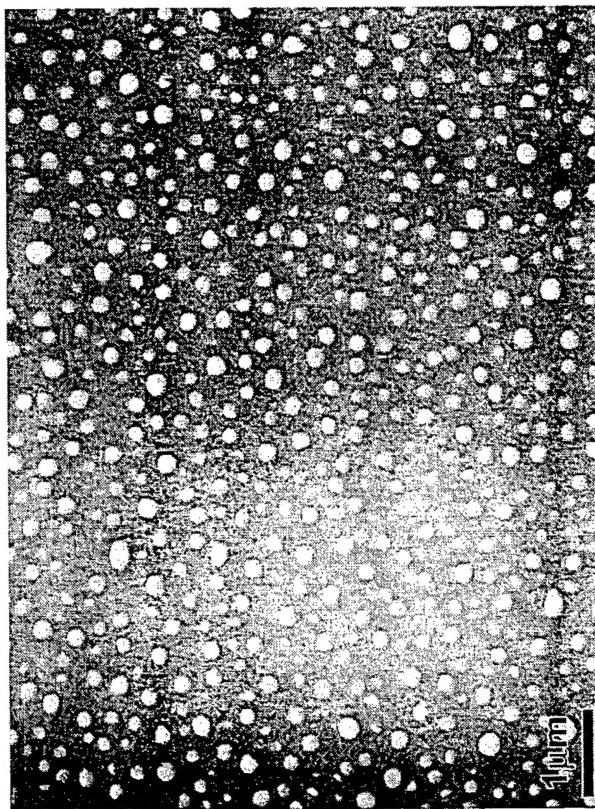
50 wt % Cp<sub>7</sub>T<sub>8</sub>Styryl in 2 million mol. wt. Polystyrene



**TEM image shows significant decrease in size of crystallites**

## POSS Blends - Miscibility

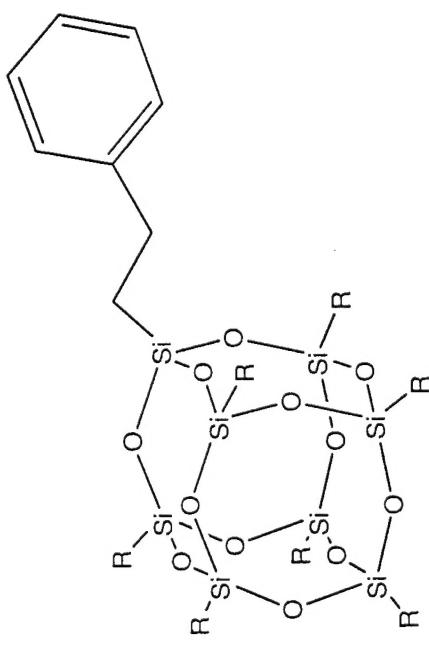
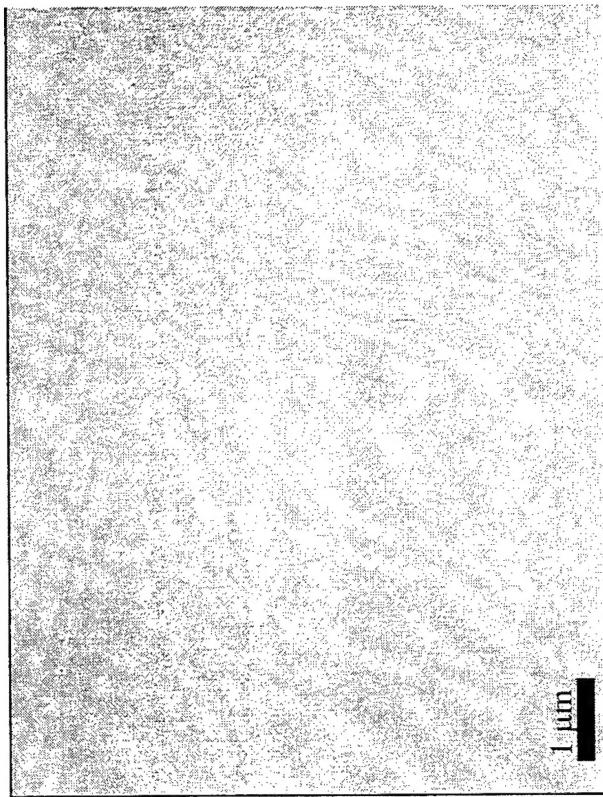
50 wt % Styrenyl<sub>8</sub>T<sub>8</sub> in 2 million mol. wt. Polystyrene



- White domains represent pure polystyrene (process issue)
- Grey domains represent miscible POSS/polystyrene
- Black dots are POSS crystallites (<100 POSS molecules)
- 30% increase in surface hardness of the material

## POSS Blends - Miscibility

50 wt % Phenethyl<sub>8</sub>T<sub>8</sub> in 2 million mol. wt. Polystyrene



- Demonstrated Complete Miscibility!!
- Grey domains represent miscible POSS/polystyrene
- Black dots are POSS crystallites (<100 POSS molecules)



## Conclusions

- The organic side groups on the POSS molecule are extremely important in determining the solubility of the POSS in polystyrene
- The addition of the more soluble styrenyl POSS into styrene leads to an increase in surface hardness without adversely affecting polymer properties
- POSS can be thought of as functionalized silicas with the side groups acting as solubility enhancers



## Acknowledgements

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- Dr. Charles Lee, AFOSR (Funding)